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(54) Title: SOLDER PASTE UTILIZING AROMATIC CARBOXYLIC ACIDS (57) Abstract			
<p>A solder paste that maintains its original viscosity over time and avoids rheology deterioration is disclosed. The invention includes coating solder alloy particles with an aromatic carboxylic acid or a mixture of acids to protect the solder alloy from reacting chemically with the flux and maintain the paste viscosity and deposition capability. In one embodiment, solder powder is treated by soaking the solder particles in an aromatic carboxylic acid solution and heat drying. In an alternative embodiment, the coated particles are dried under vacuum. The coated solder particles are then mixed with flux and formed into a paste. The aromatic carboxylic acid coating, by bonding strongly to the metal powder surface, serves as a non-soluble protective barrier against flux at ambient temperature and a flux-aid at elevated temperature.</p>			
<pre> graph TD A["SOLDER ALLOY POWDER (40In40Sn20Pb) (STEP 30)"] --> B["DISSOLVE IN (Et2O) (STEP 20)"] C["SOLID BENZOIC ACID (STEP 10)"] --> B B --> D["SOAKING (STEP 40)"] B --> E["DRYING (STEP 50)"] </pre>			

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SOLDER PASTE UTILIZING AROMATIC CARBOXYLIC ACIDS

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FIELD OF THE INVENTION

This invention relates to solder pastes, and more particularly to a solder paste formulation incorporating a solder powder coated with an aromatic carboxylic acid.

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BACKGROUND OF THE INVENTION

Solder is a fusing material used to unite adjacent surfaces of less fusible metals. Most solder alloys are combinations of tin and lead. The tin and lead alloy often additionally includes other low melting metals such as cadmium, bismuth, silver, and antimony. The solder alloy may be made into many shapes and forms, such as wire, bar, foil, spheres, ring, or paste.

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During soldering, a mechanical and/or electrical connection between two or more parts is obtained. Typically, the solder is melted by heating it above its melting temperature. The molten solder flows or is applied to the desired locations, and is resolidified upon cooling. If properly made, a good solder joint provides electrical conductivity, mechanical strength, ease of manufacture, and simplicity of repair.

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Flux is typically used during the soldering process to improve the quality of the soldered connection by removing surface compounds, reducing surface tension of the molten solder alloy, and preventing oxidation of the metals contained in the solder. Most inorganic fluxes are a combination of salts and acids dissolved in water with a wetting agent. Organic fluxes, on the other hand, include water-insoluble rosins or water soluble organic acids. Rosin flux is inert, noncorrosive, and nonconductive in the cold solid state, but becomes active in removing tarnish or surface oxidation when hot. After soldering, flux residues and oxides are typically removed with a combination of

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alcohols and chlorinated solvents, or water, if the flux is water-soluble.

5 Although solder may take many forms, soft solder pastes are used in industry primarily because pastes are more readily adapted to automated manufacture and screen-printing on a substrate than are the more conventional and manually effected solder forms. Solder paste is a mixture of pre-alloyed solder powder and flux vehicle. The composition of the solder pastes of the prior art has been largely influenced by the prior formulations of fluxing or surface 10 preparing agents. These preparing agents have included substantial portions of organic acids, or organic halide salts, such as pyridine hydrochloride, triethanolamine hydrobromide, and the like.

15 The methods of automated paste deposition on a substrate typically include roller coating, pin transferring, dispensing, and screen/stencil printing. During the course of applying the paste to the target parts, the rheology (i.e., the deformation and flow) of the solder paste is the 20 primary parameter dictating the extent of success for deposition process.

25 Although the compatibility between the solder paste and metal parts being soldered often affects the ability of the solder to form a good bond with the parts, the interaction between the flux and the metal powder surface very often deteriorates the rheology of the solder paste, and consequently the deposition capability of the solder paste. A typical phenomenon is that the viscosity increases with time, particularly during the paste deposition process. This 30 is mainly due to the fact that during deposition, the solder powder particles constantly rub against each other, promoting the exposure of fresh powder surface which in turn results in cold welding of the powder. In addition, interaction of fresh powder structure with flux promotes further formation of metal salts. These salts typically are higher in 35

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molecular weight than the unreacted fluxes, and accordingly will also contribute to the viscosity increase of the paste.

Numerous patents have issued disclosing solder and flux combinations in paste form (see, for example, U.S. Pat. Nos. 5,196,070 to Ables et al.; 5,141,568 to Turner et al.; 4,654,545 to Middlestadt; and 4,557,767 to Hwang). U.S. Pat. No. 4,298,407 to Taylor discloses a flux coating for solder alloy particles that has the ability to dissolve tin oxides and effectively clean the surface of tin-containing solder powders. However, this coating formulation contains flux that is in direct contact with the surface of the solder powder particles and does not protect the surface of the solder powder from reacting with the flux. Therefore, interactions between the flux and the metal surface of the powder particles would be expected to deteriorate the rheology of the solder paste and increase its viscosity over time.

SUMMARY OF THE INVENTION

The present invention is a solder paste that maintains its original viscosity over time, and avoids rheology deterioration common in the solder and flux formulations of the prior art. The invention includes coating solder alloy particles with an aromatic carboxylic acid to protect the solder alloy from the flux and maintain the paste viscosity and deposition capability.

In one embodiment, solder powder is treated by first soaking the powder in an aromatic carboxylic acid solution, followed by drying with either heat or vacuum or both. The aromatic carboxylic acid coating, bonded strongly to the metal powder surface, serves as a non-soluble protective barrier against flux at ambient temperature and a flux-aid at elevated temperature.

In another embodiment, a solder paste is formed by mixing solder powder particles coated with an aromatic carboxylic acid with a flux material. The aromatic

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carboxylic acid that coats each particle of the paste acts as a barrier to prevent the flux from interacting with the solder metals and increasing the viscosity of the paste.

DESCRIPTION OF THE DRAWINGS

5 The invention will be more fully understood from the following detailed description taken in conjunction with the accompanying drawings in which:

Fig. 1 is a flow diagram depicting the production of the solder paste of the invention.

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DETAILED DESCRIPTION OF THE INVENTION

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A solder paste of the present invention includes solder powder particulates pre-coated with a layer of aromatic carboxylic acids and blended with a flux. Pre-coating the solder powder particles with an aromatic carboxylic acid maintains the rheological properties of the solder paste and prevents viscosity increases common in solder paste formulations of the prior art.

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Solder powder used in the present invention may include an alloy of any combination of Sn, Pb, In, Bi, Sb, Ag, Au, Cd, Ga, Cu, Zn, or any other elements commonly employed for soldering purpose. Examples of solder alloys include 63% by weight of tin and 37% by weight of lead (designated 63Sn37Pb), 62% by weight of tin, 36% by weight of lead, and 2% by weight of silver (designated 62Sn36Pb2Ag), 50% by weight indium, and 50% by weight lead (designated 50In50Pb), and 52% by weight indium, and 48% tin (designated 52In48Sn). Other combinations of metals are also possible and may be formulated for the joining of parts of preselected composition.

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The solder powder typically comprises solder particulates having a predetermined mesh range. Examples include mesh sizes ranging from -200/+325 or -325/+500 mesh. Typically, a finer mesh range is favored for applications involving smaller aperture printing processes.

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5 To prepare the solder powder for manufacture into a solder paste, the solder powder is first soaked in a solution of aromatic carboxylic acids, such as benzoic acid, salicylic acid, phthalic acid, or the like in a suitable solvent. Use of solvent will allow formation of a thin, even coating of the acid on the surface of the powder. The coating thickness can be controlled by regulating the acid concentration in the solution. The solvent used for preparation of the solution can be organic or inorganic, however a relatively high 10 volatility is preferred for facile removal of the solvent when the coated particles are dried. Organic solvents exhibiting a boiling point no higher than 60°C, such as diethyl ether or acetone, are preferred. Suitable solvents include diethyl ether, acetone, or any other solvent that is 15 capable of dissolving solid aromatic carboxylic acid.

20 The minimum volatility desired is dictated by the solder alloy melting temperature and the drying conditions desired. In order to prevent the powder from melting during the drying process, the drying temperature should be considerably lower than the melting temperature of the solder alloy. In general, the lower the melting temperature of the alloy or 25 the lower the desired drying temperature, the higher the minimum volatility of the solvent should be. The proper selection of volatility will achieve complete vaporization of the solvent in a reasonable time while maintaining the coated solder powder particles in a non-melted state.

30 The concentration of the aromatic carboxylic acids in the solution may vary from very low (e.g., 5 mM) to relatively high (e.g., 500 mM), depending on the coating thickness desired on the solder alloy particles. In general, the thicker the coating, the higher the protection efficiency. However, the time needed for sieving processes after drying the powder also increases with increasing 35 coating thickness. Typical coating thickness range from several molecular layers to few hundred molecular layers.

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Preferably, coating thicknesses will range from approximately 50 to approximately 200 molecular layers.

5 The amount of aromatic carboxylic acid needed can be calculated from the solder powder surface area exposed to the acid solution. Variables such as quantity of solder powder to be treated, average particle size, molecular weight and density of the acid, and approximate molecular shape of an acid molecule, such as a cube or sphere, may be used to arrive at a desired amount.

10 The total aromatic carboxylic acid coating thickness, T, can be calculated from the equation:

$$T = \frac{w_a/d_a}{\frac{w_s/d_s}{\frac{4}{3}\pi r_s^3} \cdot 4\pi r_s^2}$$

(Equation 1)

where: w_a = total weight of aromatic carboxylic acid used in preparing the acid solution;

15 d_a = density of the aromatic carboxylic acid;
 w_s = total weight of the solder powder used;
 d_s = density of the solder alloy;
 r_s = average radius of the solder powder particles.

20 Assuming that the molecular shape of the aromatic carboxylic acid is a cube, a single molecular thickness, T_a , of this acid can be calculated by the equation:

$$T_a = \left[\frac{M_a/d_a}{6.02 \times 10^{23}} \right]^{1/3}$$

(Equation 2)

where: M_a = molecular weight of the aromatic carboxylic acid.

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Hence, the number of molecular layers, N_a , of aromatic carboxylic acid can be calculated:

$$N_a = \frac{T}{T_a}$$

(Equation 3)

During exposure to the solution, the aromatic carboxylic acids bond strongly to the powder surface by forming metal salts with the surface of the solder particles. This crystalline layer provides protection from the flux used in subsequent soldering operations and maintains the viscosity and rheology characteristics.

The powder slurry formed from the solder particles and the aromatic carboxylic acid solution is subsequently dried either in a ventilated oven or under vacuum. In the case of ventilated oven drying process, purging the oven with inert gas, such as argon or nitrogen, is recommended to avoid oxidation reactions. The temperature of the oven should be considerably lower than the melting temperature of the solder alloy in order to prevent excessive agglomeration. In one embodiment, a drying temperature of 60°C is considered adequate when dealing with solder alloy 40In40Sn20Pb which has a melting temperature range from 121-130°C. A preferred drying condition is a combination of vacuum and gentle heating treatment, with the heating temperature being at least 50°C lower than the melting temperatures of solder alloy and aromatic carboxylic acids. The powder obtained is then sieved through screens with preselected mesh dimensions in order to ensure an adequate particle size distribution. After the sieving process, the discrete solder powder particles are then ready to be mixed with flux to produce solder paste.

Coating solder particles with aromatic carboxylic acids confers several advantages to the formulations of the resulting solder pastes. Aromatic carboxylic acids typically have a relatively high acidity due to resonance stabilization

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from the aromatic ring. The high acidity permits rapid interaction and enhanced bonding between the aromatic acids and the metal powder surface to form metal salts. This metal salt layer serves as a good base for further deposition of aromatic carboxylic acid molecules during the drying process.

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In addition, aromatic carboxylic acids typically have a relatively high melting point, due to the strong crystallization force caused by high molecular structural regularity. For example, benzoic acid has a melting point of approximately 122°C. The highly crystalline coating structure of the aromatic carboxylic acids not only eliminates the agglomeration problem associated with many non-crystalline, sticky coating materials, such as rosin, but also provides a strong solvent resistance against the flux in the solder paste. The aromatic carboxylic acid coating prevents the solder powder particles from physically and chemically interacting with the flux, thereby reducing metal salt formation and subsequent rheology deterioration and viscosity increases.

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Upon heating and reflow, the aromatic carboxylic acids coating readily melts or dissolves in the flux, reacts, and eliminates the metal oxide as a flux-aid.

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In an illustrative embodiment, depicted in Fig. 1, 0.16 g of solid benzoic acid (step 10) was dissolved in 10 ml of diethyl ether (step 20) to prepare an 87 mM benzoic acid solution. 86 g solder alloy powder composition of 40% by weight indium, 40% by weight tin, and 20% by weight lead (40In40Sn20Pb) with a mesh range of -200/+325 (step 30) was soaked (step 40) for 60 minutes with occasional stirring in the benzoic acid solution to coat the solder alloy particles.

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The powder slurry was dried (step 50) for approximately 16 hours in a ventilated oven at approximately 60°C. The benzoic acid solution provided a coating thickness of approximately 100 molecular layers on each solder particle.

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EXAMPLE 1

In an illustrative embodiment, depicted in Fig. 1, 0.16

g of solid benzoic acid (step 10) was dissolved in 10 ml of

diethyl ether (step 20) to prepare an 87 mM benzoic acid

solution. 86 g solder alloy powder composition of 40% by

weight indium, 40% by weight tin, and 20% by weight lead

(40In40Sn20Pb) with a mesh range of -200/+325 (step 30) was

soaked (step 40) for 60 minutes with occasional stirring in

the benzoic acid solution to coat the solder alloy particles.

The powder slurry was dried (step 50) for approximately 16 hours in a ventilated oven at approximately 60°C. The benzoic acid solution provided a coating thickness of approximately 100 molecular layers on each solder particle.

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The number of molecular layers was estimated by assuming the "footprint" of an acid molecule to be 6 Å in diameter. The dried powder was then sieved through a 200 mesh screen. The selected powder was blended with a rosin-based moderately activated (RMA) flux (50% by weight rosin, 41% by weight glycol solvent, 4% by weight succinic acid, 5% by weight castor oil derivative) to make solder paste with 88% metal load by weight. The solder paste formulated by this method can then be used for reflow soldering or other soldering procedures.

Although the invention has been shown and described with respect to an illustrative embodiment thereof, it should be appreciated that the foregoing and various other changes, omissions and additions in the form and detail thereof may be made without departing from the spirit and scope of the invention as delineated in the claims.

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CLAIMS

We claim:

1. A solder paste formulation comprising:

a flux; and

5 solder powder particles coated with at least one aromatic carboxylic acid.

2. The solder paste formulation of claim 1, wherein said solder powder particles are chosen from the group consisting of Sn, Pb, In, Bi, Sb, Ag, Au, Cd, Ga, Cu, Zn, and combinations thereof.

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3. The solder paste formulation of claim 1, wherein said at least one aromatic carboxylic acid comprises benzoic acid, salicylic acid, and phthalic acid.

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4. The solder paste formulation of claim 1, wherein said at least one aromatic carboxylic acid coating is approximately 5 to approximately 500 molecular layers thick.

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5. A method of making solder paste comprising the steps of:
providing solder powder particles;
providing at least one aromatic carboxylic acid;
coating said solder powder particles with said aromatic carboxylic acid to produce coated particles;
drying said coated particles; and
mixing said coated particles with a flux.

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6. The method of claim 5, wherein said solder powder particles are chosen from the group consisting of Sn, Pb, In, Bi, Sb, Ag, Au, Cd, Ga, Cu, Zn, and combinations thereof.

7. The method of claim 5, wherein said at least one aromatic carboxylic acid comprises benzoic acid, salicylic acid, and phthalic acid.

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8. The method of claim 5, wherein said at least one aromatic carboxylic acid is dissolved in a solvent, said solvent having a vaporization temperature lower than the melting temperature of said solder powder particles.

5 9. The method of claim 5, wherein said drying step includes heating.

10. The method of claim 5, wherein said drying step includes application of vacuum.

10 11. A coating for solder alloy powder comprising at least one aromatic carboxylic acid.

12. The coating of claim 11, wherein said solder alloy is chosen from the group consisting of Sn, Pb, In, Bi, Sb, Ag, Au, Cd, Ga, Cu, Zn, and combinations thereof.

15 13. The coating of claim 11, wherein said aromatic carboxylic acid comprises benzoic acid, salicylic acid, and phthalic acid.

20 14. The coating of claim 11, wherein said at least one aromatic carboxylic acid is dissolved in a solvent, said solvent having a vaporization temperature lower than the melting temperature of said solder powder particles.

15. The coating of claim 11, wherein said coating is approximately 5 to approximately 500 molecular layers thick.

16. A method of coating solder alloy powder particles comprising the steps of:

25 providing solder alloy powder particles;
providing at least one aromatic carboxylic acid dissolved in a solvent;

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soaking said solder alloy powder particles in said at least one aromatic carboxylic acid dissolved in said solvent to produce coated solder alloy powder particles; and
5 drying said coated solder alloy powder particles to remove said solvent.

17. The method of claim 16, wherein said solder powder particles are chosen from the group consisting of Sn, Pb, In, Bi, Sb, Ag, Au, Cd, Ga, Cu, Zn, and combinations thereof.

10 18. The method of claim 16, wherein said at least one aromatic carboxylic acid comprises benzoic acid, salicylic acid, and phthalic acid.

15 19. The method of claim 16, wherein said coated solder alloy powder particles are coated with approximately 5 to approximately 500 molecular layers of said at least one aromatic carboxylic acid.

20. The method of claim 16, wherein said solvent has a vaporization temperature lower than the melting temperature of said solder alloy powder particles.

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AMENDED CLAIMS

[received by the International Bureau on 9 June 1995 (09.06.95);
original claims 1, 5, 8, 11 and 16 amended; new claims 22-37 added;
remaining claims unchanged (6 pages)]

1. A solder paste formulation, comprising:
 - a flux; and
 - solder powder particles coated with at least one aromatic carboxylic acid;

5 said coating of at least one aromatic carboxylic acid forming a complex with said solder powder particles, said coating capable of maintaining the viscosity of said solder paste formulation.
- 10 2. The solder paste formulation of claim 1, wherein said solder powder particles are chosen from the group consisting of Sn, Pb, In, Bi, Sb, Ag, Au, Cd, Ga, Cu, Zn, and combinations thereof.
- 15 3. The solder paste formulation of claim 1, wherein said at least one aromatic carboxylic acid comprises benzoic acid, salicylic acid, and phthalic acid.
4. The solder paste formulation of claim 1, wherein said at least one aromatic carboxylic acid coating is approximately 5 to approximately 500 molecular layers thick.
- 20 5. A method of making solder paste comprising the steps of:
 - providing solder powder particles;
 - providing at least one aromatic carboxylic acid dissolved in a solvent;
 - coating said solder powder particles with said at least one aromatic carboxylic acid dissolved in said solvent to produce coated particles, said at least one aromatic carboxylic acid forming a complex with said solder powder particles, said at least one aromatic carboxylic acid coating capable of maintaining the viscosity of said solder paste;
 - 25 drying said coated particles; and
 - 30 mixing said coated particles with a flux.

AMENDED SHEET (ARTICLE 19)

6. The method of claim 5, wherein said solder powder particles are chosen from the group consisting of Sn, Pb, In, Bi, Sb, Ag, Au, Cd, Ga, Cu, Zn, and combinations thereof.

5 7. The method of claim 5, wherein said at least one aromatic carboxylic acid comprises benzoic acid, salicylic acid, and phthalic acid.

8. The method of claim 5, wherein said solvent has a vaporization temperature lower than the melting temperature of said solder powder particles.

10 9. The method of claim 5, wherein said drying step includes heating.

10. The method of claim 5, wherein said drying step includes application of vacuum.

15 11. A coating for solder alloy powder particles, comprising at least one aromatic carboxylic acid, said coating forming a complex with said solder powder particles, said coating capable of maintaining the viscosity of a solder paste.

20 12. The coating of claim 11, wherein said solder alloy is chosen from the group consisting of Sn, Pb, In, Bi, Sb, Ag, Au, Cd, Ga, Cu, Zn, and combinations thereof.

13. The coating of claim 11, wherein said aromatic carboxylic acid comprises benzoic acid, salicylic acid, and phthalic acid.

14. The coating of claim 11, wherein said at least one aromatic carboxylic acid is dissolved in a solvent, said solvent having a vaporization temperature lower than the melting temperature of said solder powder particles.

5 15. The coating of claim 11, wherein said coating is approximately 5 to approximately 500 molecular layers thick.

16. A method of coating solder alloy powder particles comprising the steps of:

providing solder alloy powder particles;

10 providing at least one aromatic carboxylic acid dissolved in a solvent;

15 soaking said solder alloy powder particles in said at least one aromatic carboxylic acid dissolved in said solvent to produce coated solder alloy powder particles, said aromatic carboxylic acid dissolved in said solvent and forming a metal salt complex with said solder alloy powder particles, said coating of aromatic carboxylic acid capable of maintaining the viscosity of a solder paste; and

20 drying said coated solder alloy powder particles to remove said solvent.

17. The method of claim 16, wherein said solder powder particles are chosen from the group consisting of Sn, Pb, In, Bi, Sb, Ag, Au, Cd, Ga, Cu, Zn, and combinations thereof.

25 18. The method of claim 16, wherein said at least one aromatic carboxylic acid comprises benzoic acid, salicylic acid, and phthalic acid.

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19. The method of claim 16, wherein said coated solder alloy powder particles are coated with approximately 5 to approximately 500 molecular layers of said at least one aromatic carboxylic acid.

5 20. The method of claim 16, wherein said solvent has a vaporization temperature lower than the melting temperature of said solder alloy powder particles.

21. The solder paste formulation of claim 1, wherein said complex is a metal salt.

10 22. The method of claim 5, wherein said complex is a metal salt.

23. The coating of claim 11, wherein said complex is a metal salt.

15 24. A solder paste formulation made from a process comprising the steps of:

providing solder powder particles;

providing at least one aromatic carboxylic acid dissolved in a solvent;

20 coating said solder powder particles with said at least one aromatic carboxylic acid dissolved in said solvent to produce coated particles, said at least one aromatic carboxylic acid forming a complex with said solder powder particles, said coating of aromatic carboxylic acid capable of maintaining the viscosity of said solder paste formulation;

25 drying said coated particles; and

mixing said coated particles with a flux.

25. The solder paste formulation of claim 24, wherein said complex is a metal salt.

AMENDED SHEET (ARTICLE 19)

26. The solder paste formulation of claim 24, wherein said solder powder particles are chosen from the group consisting of Sn, Pb, In, Bi, Sb, Ag, Au, Cd, Ga, Cu, Zn, and combinations thereof.

5 27. The solder paste formulation of claim 24, wherein said at least one aromatic carboxylic acid comprises benzoic acid, salicylic acid, and phthalic acid.

10 28. The solder paste formulation of claim 24, wherein said solvent has a vaporization temperature lower than the melting temperature of said solder powder particles.

29. The solder paste formulation of claim 24, wherein said drying step includes heating.

30. The solder paste formulation of claim 24, wherein said drying step includes application of vacuum.

15 31. A solder particle formulation made from a process comprising the steps of:

providing solder powder particles;

providing at least one aromatic carboxylic acid dissolved in a solvent;

20 coating said solder powder particles with said at least one aromatic carboxylic acid dissolved in said solvent to produce coated particles, said at least one aromatic carboxylic acid forming a complex with said solder powder particles, said coating capable of maintaining the viscosity of said solder particle formulation; and

25 drying said coated particles.

32. The solder particle formulation of claim 31, wherein said complex is a metal salt.

AMENDED SHEET (ARTICLE 19)

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33. The solder particle formulation of claim 31, wherein said solder powder particles are chosen from the group consisting of Sn, Pb, In, Bi, Sb, Ag, Au, Cd, Ga, Cu, Zn, and combinations thereof.

5 34. The solder particle formulation of claim 31, wherein said at least one aromatic carboxylic acid comprises benzoic acid, salicylic acid, and phthalic acid.

10 35. The solder particle formulation of claim 31, wherein said solvent has a vaporization temperature lower than the melting temperature of said solder powder particles.

36. The solder particle formulation of claim 31, wherein said drying step includes heating.

37. The solder particle formulation of claim 31, wherein said drying step includes application of vacuum.

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AMENDED SHEET (ARTICLE 19)

1/1

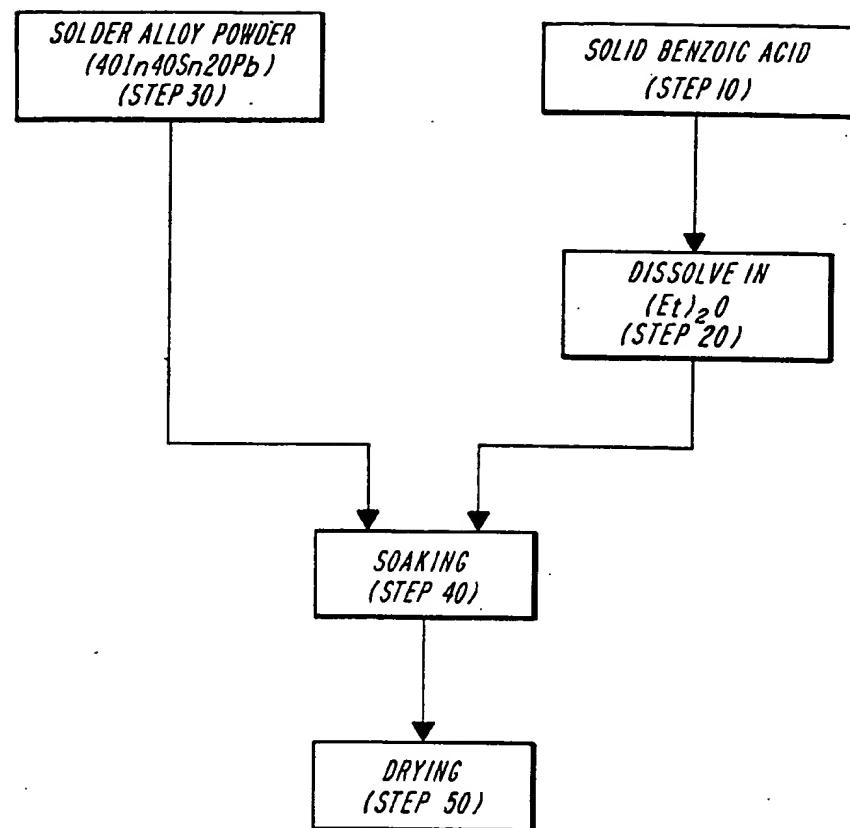


FIG. 1

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US95/01078

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :B23K 35/34
US CL :148/24

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 148/23, 24, 25

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	U.S.A, 5,092,943 (DAVIS ET AL) 03 March 1992. See entire document.	1-20
A	U.S.A, 5,041,169 (ODDY ET AL) 20 August 1991. See entire document.	1-20
A	U.S.A, 3,740,831 (JORDON ET AL) 26 June 1973. See entire document.	1-20
A	U.S.A, 3,617,345 (BROWN ET AL) 02 November 1971. See entire document.	1-20
A	U.S.A, 3,235,414 (MARKS) 15 February 1966. See entire document.	1-20
A	U.S.A, 3,264,146 (MARKS) 02 August 1966. See entire document.	1-20

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Date of the actual completion of the international search	Date of mailing of the international search report
16 MARCH 1995	10 APR 1995
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	Authorized officer <i>Peter D. Rosenberg</i> PETER D. ROSENBERG
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Form PCT/ISA/210 (second sheet)(July 1992)*